

# Pretreatment of digester feed to increase digestibility

## Effect of combined heat and vacuum extrusion

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# 1 Summary

As part of the PPS Kleinschalige bioraffinage project (WP4), a test was performed to evaluate the efficacy of a combined treatment of heat and vacuum extrusion of a 50-50 wt% mixture of cow manure and maize on the small scale anaerobic digestibility of said mixture. No positive effect on the final amount produced, production speed, or methane content could be determined.



## 2 Introduction

Livestock manure may be digested in order to produce biogas, a mixture of mostly methane and carbon dioxide. The biogas can be burned in a CHP (Combined Heat and Power) installation for the production of electricity and heat, or it can be purified to transport fuels such as CNG or LNG (Compressed or Liquefied Natural Gas, respectively). To increase biogas yields, coproducts are usually added to the manure, resulting in codigestion. These coproducts usually are agricultural by-product streams, such as left over ensilaged maize fodder, or low quality/unsold batches of potato or sugar beet. In the case of coproducts that are high in lignocellulose, such as maize silage, a pretreatment of the material may increase the digestibility by increasing the accessibility of the material to the enzymes and microorganisms in the digester.

In this report, an experiment is described in which a pretreatment consisting of a heat treatment followed by vacuum extrusion (BetaProcess, DSD, the Netherlands) is tested for efficacy. If the pretreatment is effective, it should result in an increased digestibility, which is tested in a standardised small scale digestion system, by LeAf in Wageningen. Increased digestibility is defined as markedly more biogas per amount of substrate, or an increase in the speed of biogas formation.





## 3 Materials and Methods

### 3.1 Raw materials

Fresh cow manure was used from the Wageningen UR dairy farm situated at the Runderweg, Lelystad, the Netherlands. Maize silage was taken from the stock normally used as feed at said farm, as well as coproduct for the ACRRES pilot scale digester at the same location. A mixture was made consisting of 50 % by weight of each component; 8.8 kg of each. The mixture was left to soak for half an hour, before dividing the mixture in two halves, in covered buckets. One half was left 'untreated' until the start of the digestibility test later that afternoon. The other half was treated.

### 3.2 Treatment

The treatment consisted of two consecutive steps:

1. About 1 to 1.5 kg of mixture was heated to 70 °C in a lid-covered pan over an electric stove, while continuously shaking horizontally as well as vertically. Manual stirring was also applied while checking temperature at intervals or around 1 minute. When 70 °C was reached, the pan was emptied in a bucket for the next treatment, while still hot.
2. About 0.5 L at a time, the material was fed through the vacuum extrusion device, of which the feeding tube was heated to 70 °C. The device is called the mini-Beta and build by DSD, the Netherlands, to mimic their larger Betaprocess. The smaller scale device used in this test consists of a vessel of approximately 50 L, which is continually kept under a vacuum; close to 0 bar as measured by an included manometer. Via a heated lock feeding system, about 1 L of material can be very quickly brought in the low pressure vessel. The temperature of the material going into the lock was measured to be about 50-52 °C.

The resulting 'treated' material was collected in a covered bucket and immediately transported from Lelystad to LeAf in Wageningen, where the digestibility test was started immediately.

### 3.3 Anaerobic digestibility

Digestibility tests and analyses at LeAf were started on the same day as the treatment was performed. The description of the tests, analyses, and results are translated from the LeAf report (nr 14981).

#### 3.3.1 Characterisation analyses

A characterisation was performed, focusing on dry matter, volatile matter, pH, and volatile fatty acids. All analyses were performed on duplicate samples.

#### *pH*

pH was measured using an electrode that was calibrated daily.

#### *Volatile fatty acids*

The volatile fatty acid content of the dissolved fraction was determined by gas chromatography. Sample pretreatment consisted of dilution of the substrate, 10 minute centrifugation at 10000 rpm, and diluting the supernatant with 3% formic acid. This analysis includes volatile fatty acids with a chain length of 2 to 5 carbon atoms (acetic, propionic, butyric, and valeric acid). Detection limit for each fatty acid is 50 mg/L.)

#### *Gas production and –composition*

The volume of produced biogas was determined using a system that measures pressure. The fractions of methane and carbon dioxide are determined by gas chromatography.

#### *Dry matter and volatile matter content*

The dry matter content and volatile matter content were determined by the LeaF standard method, drying samples at 105 °C and calcination at 550 °C.

### 3.3.2 Digestibility tests

Tests were run in triplicate, in 2 L bottles, using a total liquid volume of 400 mL. For each bottle, 14 g (wet weight) of treated/untreated material was used. For inoculation, a mixture of granular sludge and digested cow manure was used. A control run using only inoculate was performed to be able to correct for endogenous biogas production by the inoculate. Nutrients, trace elements, and phosphate buffer were added to all bottles. Tests have been performed at 30 °C, under continuous stirring using a shaking tray (100 rpm). Before starting the tests, the gas phase in all bottles was flushed with nitrogen. Gas production during the test was monitored regularly by measurement of pressure. At the beginning and end of the test, pH was measured. At the end of the test, biogas composition and concentration of volatile fatty acids was analysed. The test ran for 35 days.

## 4 Results

### 4.1 Characterisation

The combined treatment of heat and vacuum extrusion seems to result in an increase in acetate concentration (Table 1). The increase in volatile fatty acid content and decrease in pH are presumed to be linked to the acetate increase. Dry matter content and volatile matter content remain unchanged.

*Table 1. Characteristics of the material used for the digestibility test.*

		Treated	Untreated
pH	-	6.9	7.5
Dry matter (DM)	g/kg	189 ± 1	184 ± 9
Volatile matter (VM)	g/kg	153 ± 1	147 ± 9
% VM of DM	%	81	80
Volatile fatty acids	gCOD/L	9.17 ± 0.03	8.17 ± 0.28
Acetate	g/L	6.28 ± 0.04	5.55 ± 0.26
Propionate	g/L	0.34 ± 0.03	0.27 ± 0.00
Butyrate	g/L	1.02 ± 0.02	0.95 ± 0.00
Valerate	g/L	0.05 ± 0.00	0.06 ± 0.00

Average values from duplicate samples, ± standard deviation

### 4.2 Digestibility test

As can be seen in Figure 1, biogas production over time for the treated and untreated material is very similar. One of the three tests using treated material lags somewhat, for unknown reasons. No positive effect of the treatment on the speed of digestion or final biogas production can be claimed from these results. Between day 3 and 6, it may seem that the treated material performs less well compared to the untreated material, but differences are small and temporary, and therefore assumed insignificant.

The final numbers for biogas production and methane content as shown in Table 2 also show no effect of the combined treatment of heat and vacuum extrusion.

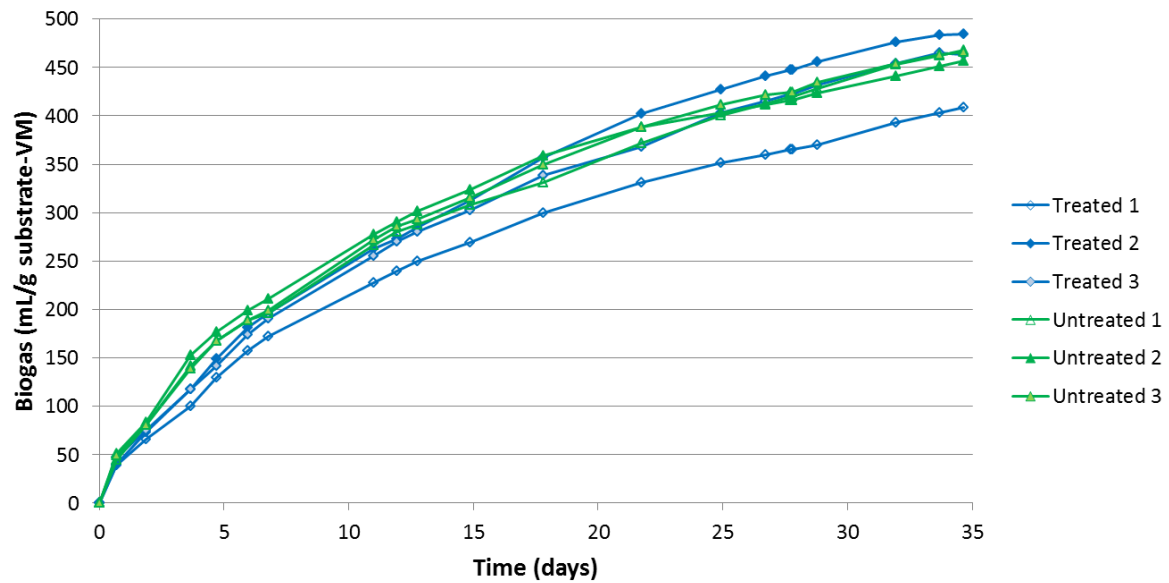


Figure 1. Biogas production (mL/g substrate-VM), measured at 30 °C.

Table 2. Biogas production and methane content, measured at 30 °C.

Sample	Biogas production		CH <sub>4</sub> content in biogas (%)
	(m <sup>3</sup> /t substrate-VM)	(m <sup>3</sup> /t substrate)	
<b>Treated</b>			
Test 1	408	62	61
Test 2	484	74	61
Test 3	463	71	61
Average	452 ± 39	69 ± 6	61
<b>Untreated</b>			
Test 1	468	69	62
Test 2	457	67	62
Test 3	466	68	62
Average	464 ± 6	68 ± 1	62

Average values from duplicate samples, ± standard deviation

## 5 Conclusion

No positive effect could be determined on the anaerobic digestibility of a mixture consisting of equal amounts by weight of fresh cow manure and maize, after a combined treatment of heat and vacuum extrusion, tested as described in this report.



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